Pesticides by GC with Nitrogen-Phosphorus Detector
EPA Method 507 Revision 2.1

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Does the laboratory have records of the [ ] initial demonstration of capability and [ ] continuing demonstrations of performance?	9.1 9.3				
Records are maintained of the method blank analyzed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method?	9.2				
If within the retention time window of any analyte of interest the LRB produces a peak that would prevent the determination of that analyte, is the source of contamination determined and the interference eliminated before processing samples?	9.2				
If an initial calibration is not performed on the day of analysis, is the calibration verified by analyses of continuing calibration verifications at a minimum of two levels with each analytical batch?	10.2.4				
Check the file names and dates. Were all calibration standards analyzed on the same day? _____ If no, why? _____					
Are sample results quantitated from the initial calibration not the continuing calibration verification?	EPA 12.1				
Is a continuing calibration verification repeated at the beginning and end of each analytical batch	EPA 10.2.4				
Are laboratory control samples (LCS-standard of known amount prepared from a source independent of calibration standards or a material containing a known amount of analyte) analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per extraction or preparation method?	EPA 9.7.1				
Is a matrix spike (sample prepared by adding a known mass of target analyte to a specific amount of matrix sample) performed at a frequency of 1 in 20 samples per matrix type prepared over time, except for analytes for which spiking solutions are not available?	EPA 9.8				
Notes					

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Method Specific Requirements					
Are one or more injections of MTBE made after the analysis of a sample containing high concentration of analytes to prevent cross contamination?	EPA 4.2				
Is a calibration curve prepared for each analyte at a minimum of 3 concentrations (5 are recommended) that bracket the working range of the detector?	EPA10.2.1				
If Merphos is to be determined is the instrument calibrated with DEF (S,S,S-tributylphosphoro-trithioate)?	EPA10.2.1				
Is the average response factor used only when the RSD ≤ 20%?	EPA 10.2.3				
If the RSD is >20%, are the response ratios used to prepare a calibration curve for each compound?	EPA 10.2.3				
Are the acceptance criteria for the continuing calibration verification standards(s) ± 20%?	EPA 10.2.4				
Are the acceptance criteria for surrogate recoveries ±30%?	EPA 10.5.1				
When using internal standards, is the acceptance criteria ±30% of the response in the daily calibration check sample?	EPA 10.6.1				
Are new control limits for the LCS and matrix spike calculated after every 5-10 measurements of the LCS using the most recent 20-30 data points?	10.7.2 10.8.2				
Is instrument performance monitored on a daily basis by the analysis of a laboratory performance check sample?	10.9				
Sample Preparation	**Analyst:** _____				
Manual Method					
Is the entire sample poured into a 2 L separatory funnel after marking the water meniscus on the side of the bottle.	11.1.1				
Is the sample fortified with surrogate standard solution and the pH of the sample adjusted to pH 7 and 100 g NaCl added to the sample and dissolved before extraction?	11.1.2 11.1.3				
Is 60 mL methylene chloride added to the sample bottle, and is the bottle sealed and shaken 30 seconds to rinse the inner walls before transferring the solvent to the separatory funnel containing the sample?	11.1.4				
Is the sample extracted by vigorously shaking the funnel for two minutes with periodic venting to release excess pressure?	11.1.4				
Is the organic layer allowed to separate from the water phase for a minimum of 10 minutes before collecting the methylene chloride extract in a 500 mL Erlenmeyer flask?	11.1.4				
Notes					

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Relevant Aspect of Standards	**Method Reference**	**Y**	**N**	**N/A**	**Comments**
Is the organic layer allowed to separate from the water phase for a minimum of 10 minutes before collecting the methylene chloride extract in a 500 mL Erlenmeyer flask?	11.1.4				
Are the sample bottle rinsed, the extraction steps repeated, and the methylene chloride extracts added to the Erlenmeyer flask with two additional 60 mL volumes of methylene chloride?	11.1.5				
Is the original sample volume determined by refilling the sample bottle to the mark and transferring the water to a 1000 mL graduated cylinder?	11.1.6				
Is the sample volume recorded to the nearest 5 mL?	11.1.6				
Automated Method					
Is the entire sample poured into a 2 L separatory funnel or tumbling bottle after marking the water meniscus on the side of the bottle?	11.2.1				
Is the sample fortified with surrogate standard and the pH of the sample adjusted to pH 7 and 100 g NaCl added to the sample before extraction?	11.2.1 11.2.2 11.2.3				
Is 300 mL methylene chloride added to the sample bottle and the bottle then sealed and shaken 30 seconds to rinse the inner walls?	11.2.4				
Is the solvent then transferred to the separatory funnel or tumbling bottle containing the sample and the container sealed and shaken, venting periodically, until no further pressure release is observed?	11.2.4				
Is the sample container resealed and placed in appropriate mechanical device and shaken or tumbled for one hour?	11.2.4				
Is complete mixing of the organic and aqueous phases observed within about two minutes after starting the mixing device?	11.2.4				
After mixing, if a tumbler was used, are the contents of tumbler bottle transferred to a 2 L separatory funnel and the phases allowed to separate at least 10 minutes before collecting the methylene chloride in a 500 mL flask?	11.2.5				
Is the original sample volume determined by refilling the sample bottle to the mark and transferring the water to a 1000 mL graduated cylinder?	11.2.6				
Is the sample volume recorded to the nearest 5 mL?	11.2.6				
Extract Concentration					
Does the laboratory use a Kuderna-Danish concentrator [other concentration devices capable of meeting the IDC and QC requirements of the method are also permitted]?	11.3.1				
Is the extract dried by pouring it through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, collecting the extract in the concentrator, rinsing the column with 20-30 mL solvent and adding the rinse to the concentrator?	11.3.2				

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If the drying column is not used, are about 5 g anhydrous sodium sulfate added to the extract and the flask swirled to dry extract and allowed to sit for 15 minutes before decanting the extract?	11.3.2				
Is the sodium sulfate rinsed two more times with 25 mL portions of methylene chloride and the rinses decanted into the concentrator with the extract?	11.3.2				
Extract Concentration					
Is the extract concentrated down to a volume of 2 mL and the before exchanging the solvent with MTBE?	EPA11.3.3				
Is the solvent exchanged by adding 5-10 mL of MTBE and reconcentrating to 2 mL?	EPA 11.3.4				
Is another 50-10 mL of MTBE added and the sample again concentrated to 2 mL?	EPA11.3.4				
Is the sample brought to a final volume of 5 mL before being transferred to a GC vial?	EPA11.3.4				
Is the capped vial refrigerated at 4° C until analysis?	EPA 11.3.5				
Chromatography					
Is the primary column DB-5, 30 m x 0.25 mm I. D. x 0.25 µm, or equivalent?	EPA 6.8.1				
Is the confirmation column DB-1701, 30 m x 0.25 mm I. D. x 0.25 µm, or equivalent?	EPA 6.8.2				
Is the helium carrier gas flow rate 30 cm/sec?	EPA 6.8.1 EPA 6.8.2				
Is the helium carrier gas flow rate 30 cm/sec?	EPA 6.8.1 EPA 6.8.2				
Is the oven temperature programmed from 60-300° C at 4° C per minute?	EPA 6.8.1 EPA 6.8.2				
Is the injection volume 2 µL in splitless mode with a 45 second delay?	EPA 6.8.1				
Is the injector temperature 250° C?	EPA 6.8.1				
Is the NPD detector temperature 300° C?	EPA 6.8.1				
Are sample components identified by comparison of the sample retention time to the retention time of a reference chromatogram?	EPA 11.5.1				
Are the retention time windows of the sample components based upon actual measurements of retention times of standards including variations over the course of a day?	EPA 11.5.2				
Notes					